

# Measurement of the fracture energy in mode I of atmospheric ice accreted on different materials using a blister test

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## Abstract

Atmospheric ice is formed when supercooled water droplets strike an object such as a tree, aircraft or wind turbine. Its microstructure and properties vary widely according to the flow and thermal conditions prevailing. The present work was conducted in the Cranfield Icing Wind Tunnel for a european project called STORM (efficient ice protection Systems and simulation Techniques Of ice Release on propulsive systeMs). It aimed at collecting data on the fracture energy of atmospheric ice on four different materials - AL2024-T3, Ti-6Al-4V, Platinum and Alexit-411 - using a blister test. This particular test, firstly introduced by Andrews and Lockington [1], have been adapted by Cranfield University to be able to test the ice adhesion in-situ while ice is still accreting on the surface making it closer to real situation. The second part of the paper will focus on the influence of different parameters like the materials ice is accreted on, the total ambient temperature, the tunnel wind speed and the cloud liquid water content which have been investigated over a few icing conditions.

*Keywords:* atmospheric ice, fracture energy, ice adhesion, blister test

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## 1. Previous work

2 Much work is published on atmospheric ice but it is unusual to find data  
3 on microstructure, toughness and unambiguous measures of the adhesion of

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## Nomenclature

$a_g$	grains size of ice
$c$	radius of the flaw
$E_i$	Young's modulus of ice
FE	Fracture energy
$h$	thickness of ice above the flaw
$k$	shape (or Weibull) parameter of the Weibull distribution
$K_{Ic}$	mode I fracture toughness
$\bar{m}$	average value
$P_c$	critical pressure needed to remove the ice
T	total temperature of the flow in the tunnel
V	tunnel wind velocity
$x$	percentage of adhesive fracture
$\Gamma$	gamma function
$\theta$	fracture energy for an adhesive fracture
$\lambda$	scale parameter of the Weibull distribution
$\nu$	Poisson's ratio of ice
$\sigma$	standard deviation
$\sigma_t$	tensile strength
$2\tau$	fracture energy for a cohesive fracture
CIWT	Cranfield Icing Wind Tunnel
LWC	Liquid Water Content
MVD	Median Volume Diameter

4 the ice to the body it has formed on. Furthermore, the capabilities of partic-  
5 ular facilities limit the range of flow and thermal conditions under which ice  
6 can be made and different studies use widely different techniques to measure  
7 the properties of the resulting ice.

8 Atmospheric ice forms due to the rapid freezing of supercooled water droplets  
9 when impinging a surface. Its particular formation results in the presence  
10 of bubbles of air trapped within the material whose amount depends on the  
11 icing conditions and effects on the material properties. With a homologous  
12 temperature higher than 0.9, the mechanical properties of atmospheric ice  
13 are highly affected by changing temperature making it an even more chal-  
14 lenging material to study.

15 Fracture energy of atmospheric ice has been rarely reported previously. To

16 the authors knowledge only Hammond [2] and Yeong et al. [3] published any  
 17 values of fracture energy of ice accreted to a substrate. They both used a  
 18 blister test similar to the one reported here. The process of making ice in  
 19 Yeong et al.'s experiments was very slow ( $5 \text{ m.s}^{-1}$ ) and only one tempera-  
 20 ture was tested ( $-20^\circ\text{C}$ ). The droplets were generated by a spray nozzle using  
 21 deionised water. Their median volume diameter (MVD) was  $20 \mu\text{m}$ . The ice  
 22 was built to a thickness of 10 mm before the mechanical test was conducted.  
 23 Each test was repeated three times for statistical purpose. Values between  
 24  $0.5$  and  $0.82 \text{ J.m}^{-2}$  were found for ice accreted on titanium alloy and between  
 25  $0.61$  and  $0.85 \text{ J.m}^{-2}$  for ice on aluminium 6061.  
 26 Hammond's experiments were conducted in a running icing tunnel at tem-  
 27 perature of  $-5$ ,  $-10$  and  $-25^\circ\text{C}$  with a cloud liquid water content (LWC) of  $0.3$   
 28  $\text{g.m}^{-3}$ , a tunnel wind speed of  $140 \text{ m.s}^{-1}$  and a droplet MVD of  $20 \mu\text{m}$ . The  
 29 type of fracture has been thoroughly reported and has been found to vary  
 from adhesive to cohesive with decreasing temperature (table 1). The only

Temperature (in $^\circ\text{C}$ )		-5	-10	-25
Ti-6Al-4V	Fracture energy ( $\text{J.m}^{-2}$ )	1.3	3.9	> 4.0
	type of fracture (% adhesive)	50	0	0
Al2024 polished	Fracture energy ( $\text{J.m}^{-2}$ )	2.1	2.8	2.4
	type of fracture (% adhesive)	80	30	0
Nickel 99.99% ground	Fracture energy ( $\text{J.m}^{-2}$ )	1.6	4.0	3.0
	type of fracture (% adhesive)	100	50	0
Stainless steel	Fracture energy ( $\text{J.m}^{-2}$ )	1.6	3.4	3.0
	type of fracture (% adhesive)	80	25	0

Table 1: Values of fracture energy reported by Hammond [2]

30 difference between material in term of type of fracture was found with Tita-  
 31 nium alloy which had a mix type of fracture even at the highest temperature  
 32 tested. Looking at the fracture energy values, they have been found to differ  
 33 largely with temperature and to a less extent with material. Nothing was  
 34 said about the number of test carried out with each material.  
 35

36 More authors have attempted to measure the tensile strength of atmospheric  
 37 ice which can be linked to the fracture energy in mode I using the Griffiths  
 38 criterion. The traditional method to measure the tensile strength, where a  
 39 sample of the material is elongated until it breaks, is challenging in case of  
 40 atmospheric ice. The difficulty is to have a good grip on the end of the spec-  
 41 imens and to measure the deformation of the sample.

42 Druez et al. [4, 5] and Tremblay [6] have studied the tensile strength of at-  
43 mospheric ice in function of ice growth parameters and the test conditions.  
44 The ice was accumulated in a cold chamber around a cylinder. This cylinder  
45 was made of two parts which were hold together by an internal screw. At  
46 the end of the accretion, the screw were removed and the cylinders were only  
47 held together by the ice. Each extremities of the cylinders were attached to a  
48 traction machine which pulled the ice at a certain strain rate. One hour was  
49 needed after the ice accretion to prepare the tensile test. Values between 0.7  
50 and 5 MPa have been obtained for the tensile strength depending on the ice  
51 growing conditions and the strain rate. The highest values were obtained at  
52 a temperature of  $-14^{\circ}\text{C}$  and a LWC of  $1.2 \text{ g.m}^{-3}$  whereas the lowest values  
53 were obtained at temperature close to melting point.

54 Laforte and Laforte [7] accreted ice on aluminium bars. The ice was made  
55 using distilled ionized water and the droplet generated had a MVD of 200  
56  $\mu\text{m}$ . After the ice accretion, the specimens were kept at the ice making tem-  
57 perature of  $-10^{\circ}\text{C}$  for one hour before the mechanical test was carried out.  
58 The iced bars were pulled by a traction machine until the ice breaks off. A  
59 strain gauge was glued to the bare side of the aluminium bar to measure the  
60 deformation and the strain rate. A load cell was used to measure the tensile  
61 force applied to the iced substrate. The tensile strength was calculated from  
62 the strain at deicing using a value of 9.9 GPa for the Young's modulus of ice.  
63 An assumption was made that the strain on the aluminium bar is the same  
64 than the strain at the ice interface. The ice was observed to either detach  
65 from the substrate in one piece or to break into several pieces. In the last  
66 case, the cracks were perpendicular to the loading direction. Values between  
67 2.8 and 5 MPa were found depending on the ice thickness with the highest  
68 value obtained for the thinnest ice deposit.

69 Mohammed and Farzaneh [8] have also grown ice around a rotating cylinder,  
70 however, they had cut an ice sample from the middle of the ice piece. The  
71 ice was grown from water sprayed, with a droplet MVD of  $40 \mu\text{m}$ , a LWC of  
72  $2.5 \text{ g.m}^{-3}$  and a temperature of  $-10^{\circ}\text{C}$ , on an aluminium cylinder rotating at  
73 1 RPM to ensure a uniform ice thickness. A lathe was used to cut the ice to  
74 avoid any crack formation. Two cups were attached to the extremities of the  
75 ice sample using freezing water and the whole was let to rest for two to three  
76 hours. A closed loop electrohydraulic machine was used to pull on the ice  
77 specimen. This method gets rid of the influence of the metal, compared to  
78 the previous ones, but needs a lot of manipulations (cutting and machining  
79 the ice samples, positioning the extensometer, etc.) which could induce pre-

80 cracks and lead to inaccurate results. Investigation of the influence of the  
81 test temperature, the wind speed and the strain rate were conducted. Strain  
82 rate was reported to be the parameter which had the most influence on the  
83 tensile strength. The tensile strength obtained was in the range from 0.9 to  
84 1.6 MPa with the highest value obtained at a test temperature of  $-15^{\circ}\text{C}$ , a  
85 wind speed of  $15\text{ m}\cdot\text{s}^{-1}$  and a strain rate of  $5 \times 10^{-5}\text{ s}^{-1}$  (which correspond to  
86 the brittle zone where the tensile strength is independent of the strain rate).  
87 The traditional tensile test gives satisfactory results but is challenging to use  
88 in case of atmospheric ice. Moreover most of the tests were conducted few  
89 hours after the ice has been made which could lead to some inaccuracy due  
90 to the relax of thermal and internal stresses. The test presented in this paper  
91 allows us to measure the fracture energy of atmospheric ice in mode I (and  
92 therefore have an idea of the tensile strength) in a running icing tunnel while  
93 the ice is still accreting to the substrate.

## 94 **2. Methodology**

### 95 *2.1. Test principle*

96 The test rig described in the present paper is based on a blister test first  
97 suggested by Andrews and Lockington [1]. It was first modified by Hammond  
98 [2] to allow its use in a running icing tunnel and then more recently to produce  
99 a more versatile test in terms of substrate material tested and to get a higher  
100 number of values for each run. The test consists of a hollow cylinder covered  
101 by a thin plastic disc (figure 1). The front surface of the cylinder was placed  
102 in the tunnel in a certain way so it was facing the spray of supercooled water.  
103 The plastic disc was maintained in position by the use of a vacuum pump  
104 which was connected to the back of the cylinder. When a significant thick-  
105 ness of ice, to allow the test to be on plane-strain condition, was accreted on  
106 the front surface of the cylinder, pressurized nitrogen was gradually applied  
107 on ice through the hole at a rate of  $10\text{ bar/s}$  (which corresponds to a strain  
108 rate of  $10^{-4}\text{ s}^{-1}$ ). The pressure needed to break off the ice was recorded by  
109 a pressure transducer and was called critical pressure,  $P_c$ . The ice can break  
110 off in three different ways: completely adhesive (substrate surface completely  
111 free of ice), completely cohesive (fracture propagating through the ice leav-  
112 ing the substrate surface covered by a layer of ice) or mixed which is partly  
113 adhesive and partly cohesive (part of the substrate surface was completely  
114 free of ice while the other part is still covered by ice). The type of fracture  
115 was estimated visually as a percentage of adhesive failure straight after the

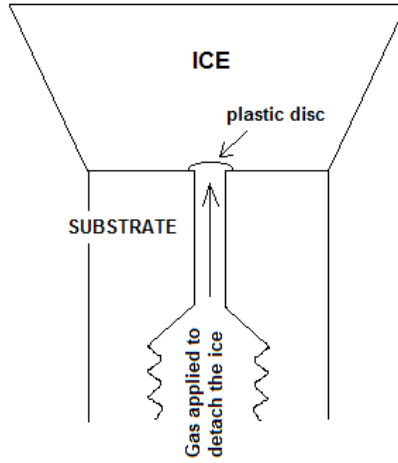


Figure 1: Scheme of the mode I test rig- the cylinder has a diameter of 30 mm, the inner hole of 4 mm and the plastic disc of 6 mm

116 ice was detached from the surface. The ice thickness need to be at least 15  
 117 mm to ensure plane strain condition which is a necessary condition in the  
 118 analytical expressions proposed by Andrews and Lockington [1]. In addition,  
 119 with an ice thickness of 15 mm or more, the error in the fracture energy due  
 120 to a misestimation of ice thickness of 2 mm would be between 0.01 and 0.02  
 121  $\text{J.m}^{-2}$ .

122 The two expressions (equations 1 and 2) established by Andrews and Lockington  
 123 [1] allow to calculate the fracture energy of ice from the critical pressure  
 124 measured during the experiments ( $P_c$ ), the Young's modulus of ice ( $E_i$ ), the  
 125 Poisson's ratio of ice ( $\nu_i$ ), the radius of the flaw ( $c$ ) and the thickness of ice  
 126 above the flaw ( $h$ ). When the type of fracture is cohesive, the fracture energy  
 127 is noted  $2\tau$  whereas when it is adhesive, the fracture energy is noted  $\theta$ . Both  
 128 notations are equivalent and represent the energy needed to create one unit  
 129 area of new crack while the fracture propagate.

$$2\tau = \frac{P_c^2 c}{E_i \times f_1} \quad (1)$$

$$\theta = \frac{P_c^2 c}{E_i \times f_2} \quad (2)$$

130 where  $f_1$  and  $f_2$  are

$$f_1 = \frac{1}{1 - \nu^2} \left( \frac{3}{32} \left[ \left( \frac{c}{h} \right)^3 + \left( \frac{c}{h} \right) \frac{4}{1 - \nu} \right] + \frac{1}{\pi} \right)^{-1}$$

$$f_2 = \frac{1}{1 - \nu^2} \left( \frac{3}{32} \left[ \left( \frac{c}{h} \right)^3 + \left( \frac{c}{h} \right) \frac{4}{1 - \nu} \right] + \frac{2}{\pi} \right)^{-1}$$

131

132 Depending on the type of fracture, cohesive or adhesive, equation 1 or 2  
 133 respectively, was used. In case of a mixed type of fracture, the percentage  
 134 of adhesive fracture,  $x$ , is visually estimated and the fracture energy, FE, is  
 135 calculated in the following way

$$FE = \frac{100 - x}{100} \times 2\tau + \frac{x}{100} \times \theta \quad (3)$$

136 The error on the estimation of the percentage of adhesive fracture is 10%  
 137 which correspond to an error on the fracture energy of about  $0.01 \text{ J.m}^{-2}$ .

138 The mode I fracture toughness,  $K_{Ic}$ , can be obtained from the fracture energy

$$K_{Ic} = \sqrt{\frac{FE \times E_i}{1 - \nu^2}} \quad (4)$$

139 From the fracture toughness, the tensile strength of a bulk of ice can be  
 140 evaluated from the following equation [9]

$$\sigma_t = \frac{K_{Ic}}{\sqrt{\pi a}} \quad (5)$$

141 where  $\sigma_t$  is the tensile strength and  $a$  the size of the defect. The largest defect  
 142 in ice is assumed to be smaller than the grain size. Therefore, the typical  
 143 size of a defect will be taken as the average grain and the tensile strength  
 144 equation becomes

$$\sigma_t = \frac{K_{Ic}}{\sqrt{\pi a_g}} \quad (6)$$

145 where  $a_g$  is the grain size.

146 The choice of the average grain size as a typical defect size can be surpris-  
 147 ing as a grain is not a real defect in a material. However, this dimension is  
 148 relatively easy to obtain from the microstructure and as default of any other  
 149 flaw dimension known within the material, the value obtained by this way is

150 assumed to give a good approximation of the tensile strength.  
151 For all the following calculations, assumptions were made that the Young's  
152 modulus and the Poisson's ratio were constant for the whole conditions tested  
153 and were taken as 8.5 GPa and 0.31 respectively. Average grains size was  
154 measured using the technique described in Pervier et al. [10].  
155 This test has numerous advantages. It has proved to be reasonably repro-  
156 ductible taken into account the brittle properties of ice which can be respon-  
157 sible for large scatter. The plastic disc is initiating the crack meaning that  
158 the flaw dimension is known. The pressure rate can be modified so different  
159 load rate can be tested. Finally, it allows to measure the adhesion of ice in  
160 a running icing tunnel while the ice is still accreting on the front face. This  
161 means that thermal stresses due to the heat release during the ice accretion  
162 process does not have the time to relax making it closer to real situation of,  
163 for example, an aircraft flying in icing conditions or a wind turbine experi-  
164 encing icing. It is worth to note that the heat released during accretion can  
165 be significant and be responsible for the introduction of non negligible shear  
166 stresses at the interface ice/substrate. The influence of the thermal effects on  
167 the test results are currently under investigation and are beyond the scope  
168 of this paper. Therefore an assumption will be made that the loading in this  
169 test is pure mode I.  
170 The dimension of the cylinder is not taken into account in equations 1 and  
171 2. The influence of this dimension has not been studied yet.

## 172 *2.2. Description of the test facilities*

173 Tests were conducted in the Cranfield Icing Wind Tunnel (CIWT). This  
174 tunnel is composed of a square test section of 760 mm in width, a fan driven  
175 by an electric motor, a return duct, a heat exchanger and a spray rake. The  
176 spray rake consists of 6 rows of nozzles with a total of 99 places for nozzles  
177 to allow the user to adapt the cloud. The nozzles bars include one pipe filled  
178 by air and within it, one pipe filled by water. By adjusting the pressure of  
179 water and air, the droplet size and the LWC of the cloud could be modified.  
180 The CIWT has the capability of recreating atmospheric icing condition for  
181 supercooled water droplet from 16 to 300 microns at temperature between 0  
182 and  $-30^{\circ}\text{C}$  and for tunnel air speed between 30 to  $110\text{ m}\cdot\text{s}^{-1}$ .

## 183 *2.3. Substrate material and testing conditions*

184 During the STORM campaign, four reference materials have been se-  
185 lected. The first two are widely used alloys in aerospace and rely on an oxide



186 film for their environmental stability. The third one is a noble metal and,  
187 therefore, does not contain an oxide layer but has free electrons at its surface.  
188 The last one is a polymer, widely used in aerospace, whose stiffness is closer  
189 to the stiffness of ice than metal.

190 - Al2024-T3 unclad and not anodized. Samples were cut from plate of  
191 1.2 mm in thickness and polish to mirror finish ( $R_a=0.01 \mu\text{m}$ )

192 - Ti-6Al-4V. Samples were cut from a plate of 2 mm thickness and polish  
193 to mirror finish ( $R_a=0.01 \mu\text{m}$ )

194 - Platinum. Samples of Al2024-T3 were prepared in the same way as  
195 described above and were then spluttered with platinum in a sputter  
196 reactor. The aluminium surface was cleaned in the sputter chamber  
197 in an Argon plasma then an adhesion promoting layer of Cr of about  
198 5 nm thickness was deposited on the aluminium surface followed by a  
199 platinum layer of about 100 nm thickness.

200 - Alexit-411 Clearcoat. This is a reference coating manufactured by  
201 Mankiewicz and used by Airbus. Samples were coated on Al2024-T3  
202 coupons slightly polished with carbide paper to obtain a flat and clean  
203 surface. The coating thickness was about  $100 \mu\text{m}$ .

204 The mirror finish condition has been obtained by the following way. First, the  
205 specimens were ground using Silicon Carbide paper starting with 180 grade  
206 grit and progressing by steps to 1200 grade grit using water as lubricant.  
207 They were then given a thorough rinse and dried with a soft cellulose based  
208 non-woven fabric (3 cycles of cleaning). The polishing was done manually  
209 using a Stuers DP plan nylon polishing mat, 3 microns diamond paste and  
210 Struers Blue (ethanol based) lubricant. The surfaces were polished three  
211 times, each time to a point where the texture from the previous polish was  
212 obliterated. The samples were washed as before, three times but using Struers  
213 Blue lubricant. They were then lapped using a Struers DP Nap as pad, 1  
214 micron diamond paste and Struers Blue lubricant until no sign remained of  
215 the polished finish. The specimens had a mirror finish with some occasional  
216 instances of slight scaring from grinding damage in less critical areas of the  
217 surface.

218 Eight icing conditions were chosen to offer a wide range of ice type from glaze  
219 to rime (table 2).

Temperature (°C)	Wind speed (m.s <sup>-1</sup> )	LWC (g.m <sup>-3</sup> )	MVD (μm)	Ice type and description
-5	80	1.0	20	glaze - transparent with
-10	50	0.8	20	evidence of runback ice
-10	80	0.8	20	mixed glaze - pretty
-5	80	0.3	20	smooth in aspect with
-5	50	0.3	20	a conical shape
-20	50	0.8	20	mixed rime - white but with transparent aspect. Presence of
-15	80	0.3	20	feathers can be spotted on side of samples. Cylindrical in shape.
-20	50	0.3	20	rime - completely opaque with feathers on side

Table 2: Icing conditions and description of the ice obtained

### 220 3. Test procedure

221 Before each test, the nozzles were checked to be sure that none of them  
222 were blocked. Eight cylinders can be placed in the tunnel at each run. They  
223 were positioned on two support bars (figure 2). Each cylinder were spaced  
224 from its closest neighbour by 10 cm (distance between the centre). To ensure  
225 a good reproductibility of the mechanical test, the surface of each cylinder  
226 was carefully cleaned with ethanol and then dried with a hot air gun. Then  
227 the whole test rig was covered. Air was sprayed from the nozzle to make sure  
228 all the water remaining in and around the nozzles was sprayed anywhere but  
229 on the specimens surface. The specimens were then uncovered and the plas-  
230 tic discs put in place. Finally the tunnel window was closed and the main  
231 fan and cooling system were started.

232 The different parameters were set (LWC, ambient temperature, tunnel air  
233 speed, droplet size) and when the temperature in the tunnel was stable, the  
234 water was sprayed. Even if an ice thickness of 15 mm was considered as  
235 sufficient, in the majority of runs (only the glaziest conditions would not  
236 allow the ice to grow towards the flow but more sidewise. In that case, 15  
237 mm was often seen as the maximum thickness reached before the ice samples



Figure 2: Mode I test rig in place in the test section of the CIWT

238 started to touched their direct neighbours in the tunnel), the ice was accreted  
239 until a thickness of 20 mm was reached before applying the pressure. With  
240 such thickness, the error due to the misestimation of ice thickness of 2 mm  
241 (typical error on the visual estimation of the ice thickness) would be below  
242  $0.01 \text{ J.m}^{-2}$ . This is considered as negligible compared to the scatter of the  
243 fracture energy values.

244 The critical pressure needed to detach the ice as well as the mode of fracture  
245 and the estimated ice thickness above the flaw were noted. It is worth to  
246 note that during the mechanical test (when the pressure is applied to the  
247 ice), the tunnel is still running and the ice is still accreting.

248 Ice is a brittle material, hence, even if care has been taken to have a re-  
249 productible test, the results present scatter. It has been found that brittle  
250 fracture follow a Weibull distribution [11]. Hence a statistical analysis was  
251 carried out using the software Statistica <sup>1</sup> in order to obtain a mean value  
252 and its standard deviation for each conditions. Parameters were chosen in  
253 order to obtain the best fit with a threshold of 0 (two parameters Weibull  
254 distribution). The Weibull parameter obtained were between 2 and 8. This  
255 falls into the family of curve where the probability of ice fracture would be

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<sup>1</sup>Statistica is a statistics and analytics software developed by StatSoft, <http://www.statsoft.com>

256 nul at  $0 \text{ J.m}^{-2}$ .

## 257 4. Results and discussion

258 In all the following, data will be presented in terms of fracture energy  
259 and comparison with previous authors will only be made using a trend with  
260 different parameters. Runs have been repeated a number of times to obtain  
261 around 10 values for each material and each icing condition. In one case ( $T=-$   
262  $15 \text{ }^\circ\text{C}$ ,  $V=80 \text{ m.s}^{-1}$ ,  $\text{LWC}=0.3 \text{ g.m}^{-3}$ ,  $\text{MVD}=20 \text{ }\mu\text{m}$ , Platinum substrate)  
263 only 5 values were obtained due to difficulties to remove the ice. An average  
264 value,  $\bar{m}$ , and a standard deviation,  $\sigma$ , were calculated using a 2 parameters  
265 Weibull distribution (equations 7 and 8) where  $\lambda$  is the scale parameter,  $k$  is  
266 the shape (or Weibull) parameter and  $\Gamma$  is the gamma function.

$$\bar{m} = \lambda \times \Gamma(1 + 1/k) \quad (7)$$

$$\sigma = \sqrt{\lambda^2[\Gamma(1 + 2/k) - (\Gamma(1 + 1/k))^2]} \quad (8)$$

### 267 4.1. Influence of material

268 Comparison of the results obtained for the four reference materials at  
269 four different icing conditions is presented on figure 3. The type of fracture  
270 is shown in the columns as a percentage of adhesive fracture (100% adhesive  
271 means fully adhesive and 0% adhesive means fully cohesive). It is displayed as  
272 the number of test that resulted in a certain type of fracture. Five different  
273 categories have been selected to simplify the graph. The colours are lighter  
274 as the type of fracture is becoming more cohesive. The crosses represents  
275 the average value of the tensile strength and the error bars one standard  
276 deviation.

277 With the two alloy materials, the ice was breaking in a predominantly ad-  
278 hesive way at the highest temperature shown and was gradually becoming  
279 cohesive as the temperature was decreased. With the platinum, even at a  
280 temperature close to melting point a mixed type of fracture was obtained.  
281 Whereas with the Alexit coating, even at low temperature, the ice was break-  
282 ing predominantly in an adhesive way. Hammond [2] studied the ice adhesion  
283 on two different grade of aluminium, Al2024 and Al7075, nickel, titanium al-  
284 loy (Ti-6Al-4V) and stainless steel. The type of fracture has been thoroughly  
285 reported and only little difference has been found between all these materials.  
286 As seen on table 1, at the highest temperature tested ice was breaking, from  
287 all material except Titanium alloy, in a predominantly adhesive way. Then,

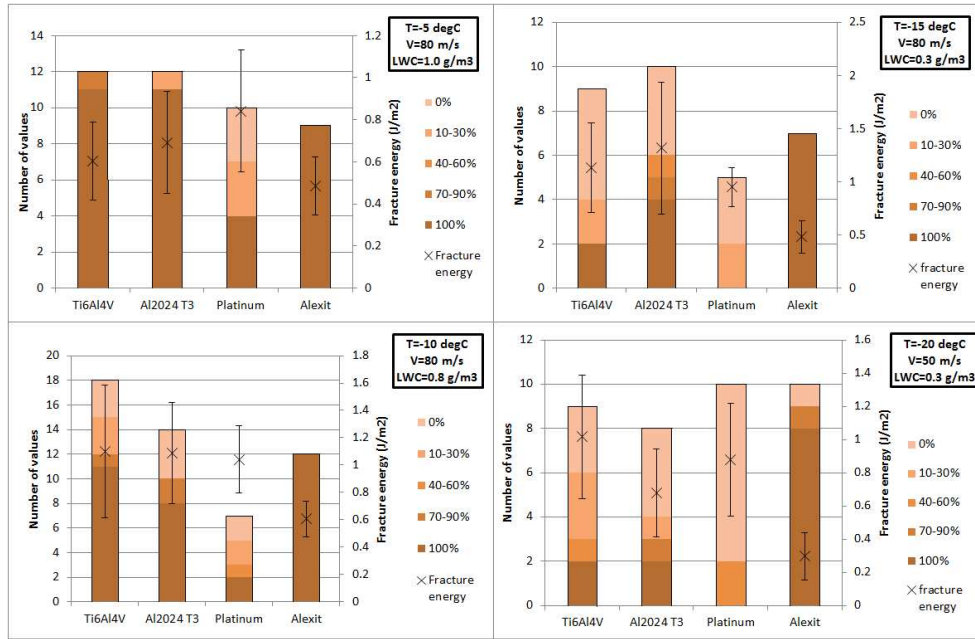


Figure 3: Type of fracture and average fracture energy values for the four reference materials at different icing conditions - the type of fracture is presented as a percentage of adhesive fracture

288 the fracture type was mixed at  $-10^{\circ}\text{C}$  and cohesive at  $-25^{\circ}\text{C}$ . For Titanium  
 289 alloy, the fracture type was mixed at  $-5^{\circ}\text{C}$  and cohesive at both  $-10^{\circ}\text{C}$  and  
 290  $-25^{\circ}\text{C}$ .

291 Alexit coating is a much softer material than Aluminium alloy, Titanium  
 292 alloy or Platinum. The standard deviation obtained with this material was  
 293 narrower than with the other materials. In general, the values obtained with  
 294 Alexit coating were lower than with the other materials meaning that the  
 295 ice adhesion was lower than on the other materials tested. On the metal-  
 296 lic materials, no clear conclusion could be drawn. In some icing conditions,  
 297 there was no difference between the three materials whereas in others, one  
 298 of them presented a slightly higher adhesion. In [2], the fracture energy val-  
 299 ues reported were lower for Aluminium alloy ( $2\text{-}3\text{ J}\cdot\text{m}^{-2}$ ) that for the other  
 300 materials ( $3\text{-}4\text{ J}\cdot\text{m}^{-2}$ ) at temperature lower than  $-10^{\circ}\text{C}$ . At a temperature of  
 301  $-5^{\circ}\text{C}$ , no difference could be found. Yeong et al. [3] reported values of simi-  
 302 lar range for aluminium and titanium at  $-20^{\circ}\text{C}$ . Values for teflon and other  
 303 hydrophobic and superhydrophobic material were at least 3 times lower.

304 4.2. Influence of total ambient temperature

Results have been presented on figure 4, sorted by material on which

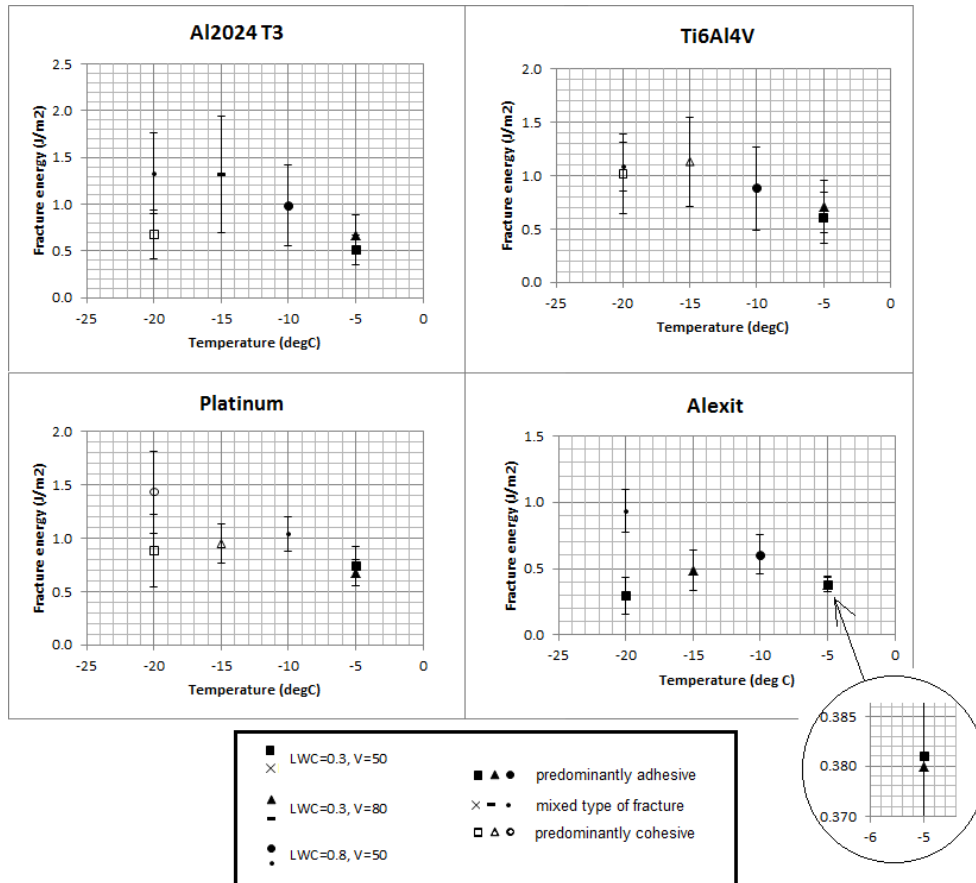


Figure 4: Influence of total ambient temperature on fracture energy of atmospheric ice accreted on each reference material (type of fracture is displayed by different symbol)

305

306 ice had been accreted to. They have been sorted in three different series  
 307 having the same LWC and tunnel wind speed to study the influence of total  
 308 ambient temperature only. Filled symbols represents fracture predominantly  
 309 adhesive, empty symbols fracture predominantly cohesive and crosses, dashes  
 310 or points mixed fracture.

311 Type of fracture, for the metallic substrates, shifted from predominantly  
 312 adhesive at -5 and -10 °C to predominantly cohesive at -15 to -20 °C. At -10  
 313 °C with the Platinum substrate, the type of fracture started to shift to mixed.

314 Whereas, for the Alexit coating, the type of fracture was predominantly  
315 adhesive in all cases except at a temperature of  $-20\text{ }^{\circ}\text{C}$  with a LWC of  $0.8$   
316  $\text{g}\cdot\text{m}^{-3}$  and a wind speed of  $50\text{ m}\cdot\text{s}^{-1}$ . A similar observation, meaning a shift  
317 from predominantly adhesive to predominantly cohesive as the temperature  
318 was decreased, was reported by Hammond [2] for all the materials he studied.  
319 All results except one (LWC= $0.3\text{ g}\cdot\text{m}^{-3}$ ,  $V=50\text{ m}\cdot\text{s}^{-1}$ , Alexit substrate)  
320 showed an increase of fracture energy as the temperature decreased. Previous  
321 authors have reported either an increase of tensile strength/fracture energy  
322 [2, 8] or a passage through a maximum between  $-10$  and  $-15^{\circ}\text{C}$  [2, 4, 5].

#### 323 *4.3. Influence of tunnel wind speed*

324 Results for the different materials have been presented on figure 5 to il-  
325 lustrate the influence of tunnel wind speed on fracture energy. Two series  
326 have been identified where only the tunnel wind speed was varied from 50 to  
327  $80\text{ m}\cdot\text{s}^{-1}$  keeping the total ambient temperature and the LWC constants.

328 As the temperature was higher than  $-10\text{ }^{\circ}\text{C}$ , the tests resulted in ice separa-  
329 tion from their substrate in a predominantly adhesive way and therefore no  
330 influence of the tunnel wind speed could be observed on the type of fracture.  
331 More experiments need to be carried out at lower temperature to have a  
332 better understanding on the influence this parameter.

333 Aluminium alloy and Titanium alloy both presented an increase of fracture  
334 energy as the speed was increased. The results obtained with the Alexit coat-  
335 ing were totally independent on wind speed. Whereas the Platinum samples  
336 presented either no dependence with tunnel wind speed (Temperature of  $-10$   
337  $^{\circ}\text{C}$  and LWC of  $0.8\text{ g}\cdot\text{m}^{-3}$ ) or a decrease of fracture energy as the speed  
338 was increased from  $50$  to  $80\text{ m}\cdot\text{s}^{-1}$  (temperature of  $-5\text{ }^{\circ}\text{C}$  and LWC of  $0.3$   
339  $\text{g}\cdot\text{m}^{-3}$ ). Druez et al. [4, 5] and Mohammed and Farzaneh [8] measured the  
340 tensile strength of ice on Aluminium alloy. They reported an increase of  
341 tensile strength as the wind speed increased up to  $15\text{-}16\text{ m}\cdot\text{s}^{-1}$  followed by  
342 a decrease as the wind speed was increased further up to  $20\text{ m}\cdot\text{s}^{-1}$  [8] or  $23$   
343  $\text{m}\cdot\text{s}^{-1}$  [4, 5].

344

#### 345 *4.4. Influence of cloud liquid water content*

346 Two series had been identified to illustrate the influence of cloud LWC  
347 for the four reference materials (figure 6). The same system of symbols have  
348 been used to represent the type of fracture as in 4.2.

349 For the Aluminium alloy, the type of fracture did not seem to depend on the

350 LWC. The type of fracture was predominantly adhesive for the series at  $-5^{\circ}\text{C}$   
351 and predominantly cohesive for the series at  $-20^{\circ}\text{C}$ . With the Titanium alloy  
352 substrate, at a temperature of  $-5^{\circ}\text{C}$ , the type of fracture remained predom-  
353 inantly adhesive as the LWC was varied from  $0.3\text{ g.m}^{-3}$  to  $1.0\text{ g.m}^{-3}$ . At a  
354 temperature of  $-20^{\circ}\text{C}$ , the type of fracture changed from predominantly cohe-  
355 sive at a LWC of  $0.3\text{ g.m}^{-3}$  to mixed at a LWC of  $0.8\text{ g.m}^{-3}$ . For the Platinum  
356 at a temperature of  $-5^{\circ}\text{C}$ , the type of fracture changed from predominantly  
357 adhesive at a LWC of  $0.3\text{ g.m}^{-3}$  to mixed at a LWC of  $1\text{ g.m}^{-3}$  while, at  
358 a temperature of  $-20^{\circ}\text{C}$ , it remained predominantly cohesive at both LWC  
359 tested. For the Alexit coating, at a temperature of  $-20^{\circ}\text{C}$ , the type of frac-  
360 ture shifted from predominantly adhesive at a LWC of  $0.3\text{ g.m}^{-3}$  to mixed  
361 at a LWC of  $0.8\text{ g.m}^{-3}$ . At a temperature of  $-5^{\circ}\text{C}$ , the type of fracture was  
362 identical for both LWC tested. Various scenarios seemed to happen with  
363 the different material and there is no obvious trend with the icing conditions  
364 tested.

365 Regarding the fracture energy, for the Titanium alloy and the Platinum sub-  
366 strates, results seemed relatively independent of LWC. For the Aluminium  
367 alloy, at a temperature of  $-5^{\circ}\text{C}$  and a wind speed of  $80\text{ m.s}^{-1}$ , no change in  
368 the fracture energy could be observed as the LWC was increased from  $0.3$  to  
369  $1\text{ g.m}^{-3}$ . On the other hand, at a temperature of  $-20^{\circ}\text{C}$  and a wind speed of  
370  $50\text{ m.s}^{-1}$ , the fracture energy almost doubled as the LWC was increased from  
371  $0.3$  to  $0.8\text{ g.m}^{-3}$ . For the Alexit coating, both series presented an increase  
372 of fracture energy as the LWC was increased but to a much larger extent at  
373  $-20^{\circ}\text{C}$  and  $50\text{ m.s}^{-1}$ . Druetz et al. [5] measured the tensile strength of ice on  
374 aluminium at two different LWC ( $0.8$  and  $1.2\text{ g.m}^{-3}$ ). They reported higher  
375 tensile strength values at the highest LWC.

## 376 5. Conclusion

377 A blister test has been successfully used to measure the fracture energy  
378 of ice in a running icing tunnel. Four reference materials have been studied  
379 under a range of icing conditions and the influence of each parameter has  
380 been investigated. In general, results were consistent with previous authors  
381 especially concerning the metallic alloys.

382 A coating Alexit-411 with a thickness of approximately  $100\text{ }\mu\text{m}$  has been  
383 found to reduce sensibly the ice adhesion compared to the metallic sub-  
384 strates. The most influencing parameter was the ambient temperature for  
385 which the fracture energy was higher at lower temperature. With the metal-



386 lic substrate, the type of fracture was also affected; a shift from adhesive  
387 fracture to cohesive fracture was observed as the temperature decreased.

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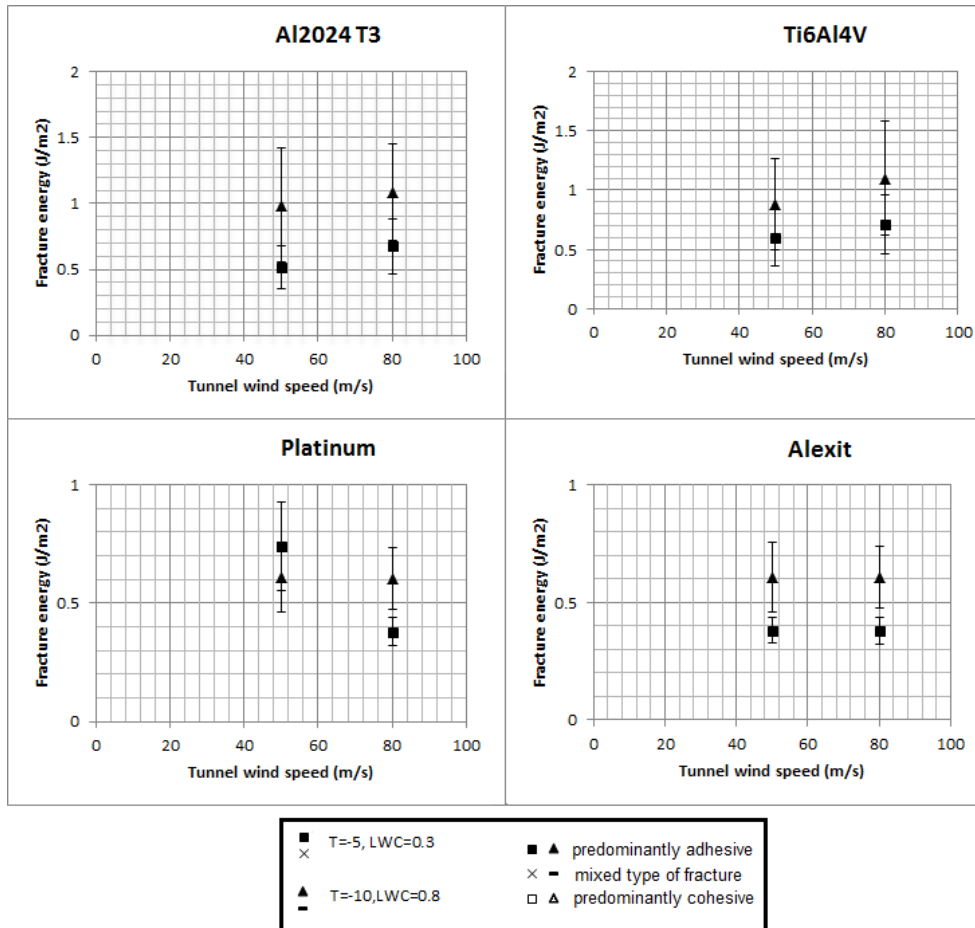


Figure 5: Influence of tunnel wind speed on fracture energy of atmospheric ice accreted on each reference material

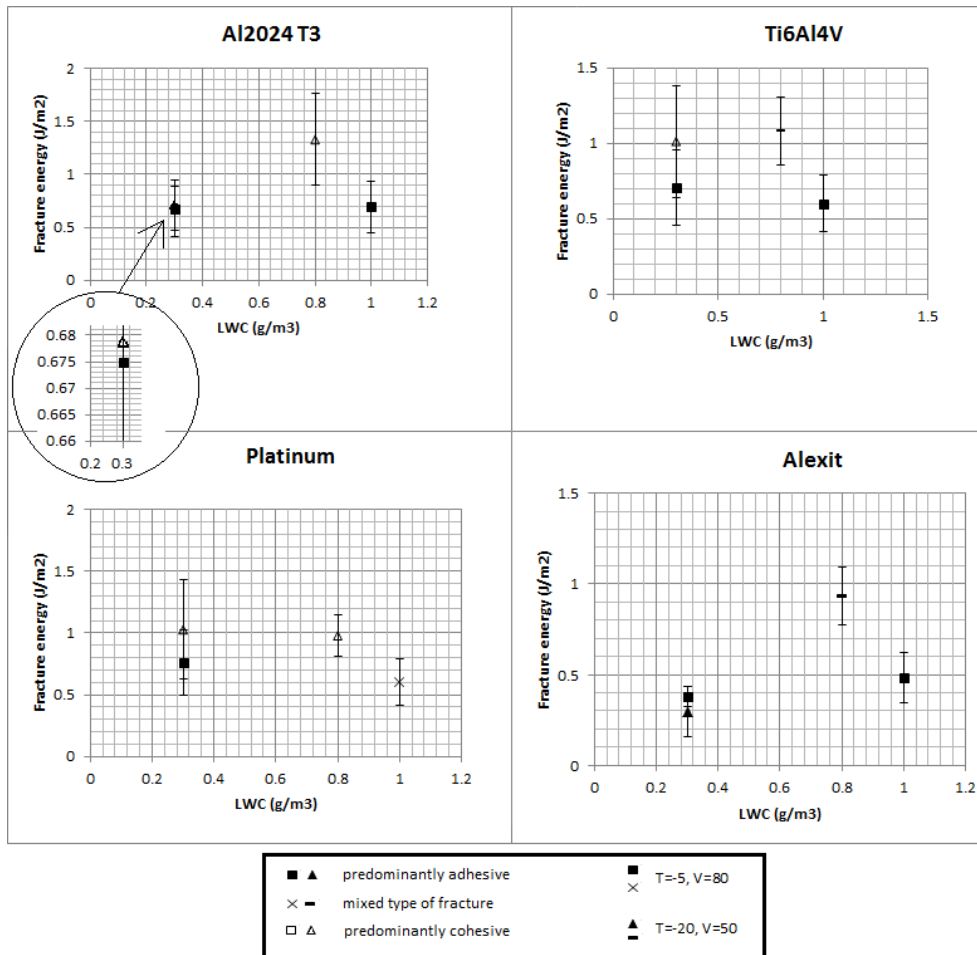


Figure 6: Influence of LWC on fracture energy of atmospheric ice accreted on each reference material